THE ELECTRICAL CONDUCTIVITY OF TELLURIUM AND OF LIQUID MIXTURES OF TELLURIUM AND SULPHUR

by

ERNEST W. JOHNSON

A THESIS

SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE OF DOCTOR OF PHILOSOPHY IN THE GRADUATE DEPARTMENT OF BROWN UNIVERSITY, 1925

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I. Introduction

The immediate purpose of the present investigation was to determine the influence of varying amounts of sulphur on the electrical conductivity of metallic tellurium. According to the literature[1] sulphur and tellurium are miscible in all proportions in the liquid state and the phase diagram indicates an absence of compounds between the two components. Since tellurium is a metallic conductor and sulphur is a non-conductor, it appeared of interest to investigate the conduction process in mixtures of these two elements as a function of composition. Solutions of metals in non-metallic solvents occur only infrequently. The only systems of this type that have been studied extensively are solutions of the alkali metals in liquid ammonia.[2] In these solutions the metallic atom appears to be ionized into a normal positive ion and an electron which is more or less associated with the solvent molecules. In a mixture, such as tellurium and sulphur, it might be anticipated that the relation between conductivity and composition would vary greatly from that of the ammonia solutions just mentioned. Tellurium itself is only very weakly electropositive and has a relatively high affinity for the electron. Sulphur has a low dielectric constant and we should expect that, in a mixture of sulphur and tellurium, the conducting power would fall rapidly with increasing concentration of sulphur. Moreover, since sulphur has a high affinity for electrons, we might expect that at higher concentrations (of sulphur) the electrons would associate themselves with sulphur atoms or molecules and that the conduction process would be of the electrolytic type. Such, in fact, has been found to be the case. Incidentally, the conductivity of pure tellurium has been measured for the solid as well as the liquid state.

The method employed in measuring the conductivity of pure tellurium as well as that of its mixtures with sulphur consisted in introducing the tellurium, or its mixture with sulphur, into suitable cells whose constants had been determined. The cells containing the materials were introduced into a thermostat whose temperature was readily varied and controlled. The mixtures were melted under an atmosphere of nitrogen. The resistance of the mixtures was measured by means of a direct current and Wheatstone bridge or an alternating current and Kohlrausch bridge.

II. Materials, Apparatus and Procedure

Tellurium. The tellurium was prepared according to the following procedure: An impure metal was dissolved in nitric acid in the presence of a

[1] Pellini: Atti. Acad. Lincei, **18** I, 701 (1909); Chikashige: Z. anorg. Chem., **72**, 109 (1911); Jaeger and Menke: **75**, 241 (1912).

[2] Kraus: J. Am. Chem. Soc., **53**, 749 (1921) and earlier papers; Kraus and Lucasse: **43**, 2531 (1921); **44**, 1941 (1922); **45**, 2551 (1923).

small amount of hydrochloric acid and evaporated to dryness. The residue was heated to decompose any traces of basic nitrate, hydrochloric acid was added and the product was again evaporated to dryness in order to remove all traces of nitric acid. This product was dissolved in strong hydrochloric acid, diluted with hot water, made slightly alkaline with ammonia and acidified with dilute acetic acid. The tellurium dioxide precipitated in this operation was thrown on a filter, washed and dried. It was then heated to 750° in an alundum boat in a quartz tube with a slow stream of oxygen passing

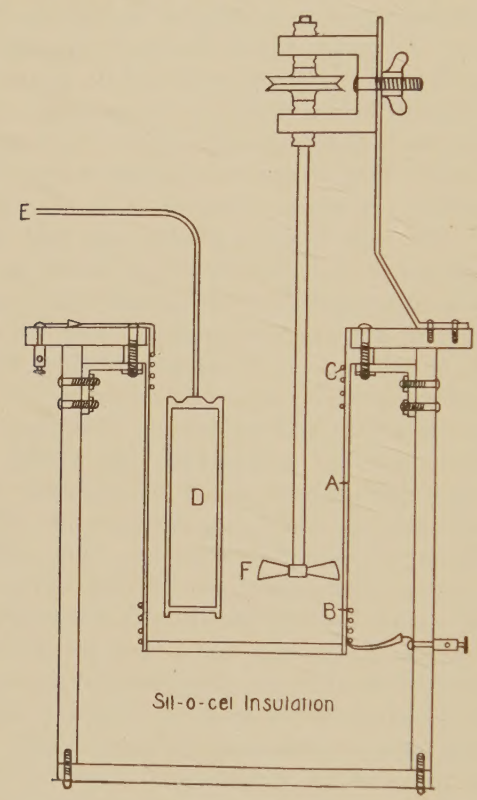

Fig. 1
Thermostat

over it. This served to remove the greater proportion of selenium dioxide present. The resulting product was dissolved in strong hydrochloric acid, the solution diluted to about 6 N, and the metal reduced by means of sulphur dioxide at a temperature of about 90°. The precipitated metal was thrown on a filter, thoroughly washed with water and finally with alcohol. After drying in a vacuum desiccator, the metal was distilled in vacuo from an alundum boat placed in a pyrex tube. The final product gave no qualitative test for probable impurities including selenium.

Sulphur. Sulphur was purified by recrystallization from freshly distilled carbon bisulphide.

The Thermostat. The resistance temperature coefficient of the various mixtures was found to be high and it was therefore necessary to provide means for accurate and convenient temperature control of the thermostat in which the cells containing the mixtures were placed. The thermostat con-

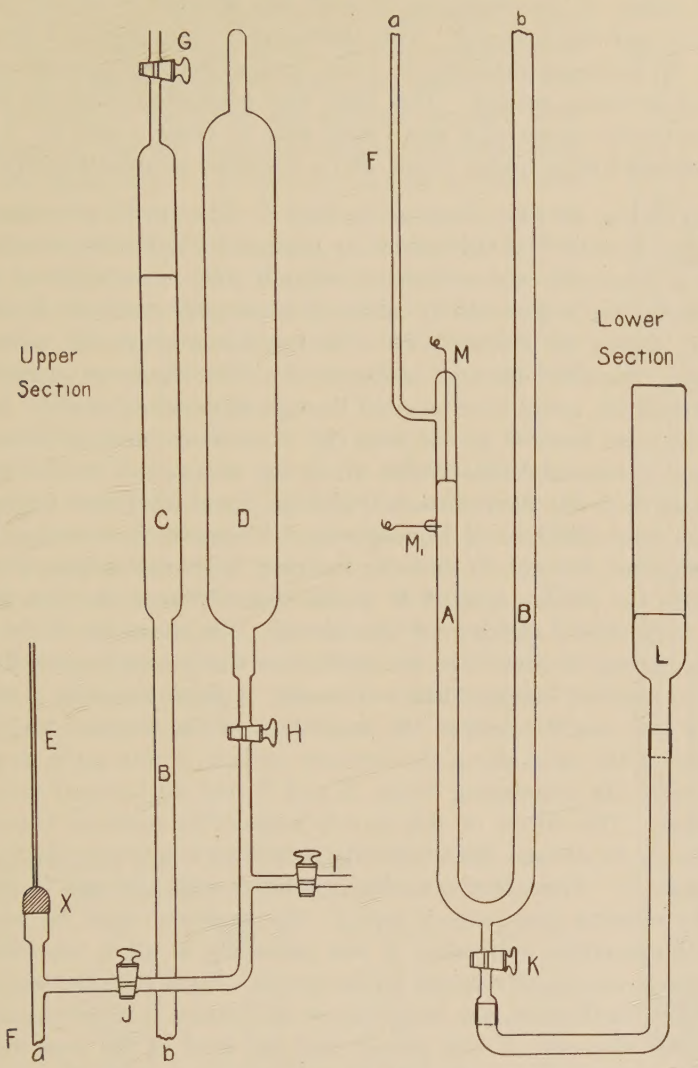

FIG. 2
Temperature Regulator

sisted of a welded metal pot *A* (Fig. 1) in which a low melting lead-tin alloy was employed as thermostatic liquid. The pot was wound on the outside with nichrome wire *B*, a current through which served to compensate for radiation loss or to heat up the thermostat initially. The pot was lagged with sil-o-cel contained in an asbestos board box. The liquid in the thermostat was thoroughly stirred by means of a motor-driven propellor *F*. Within

the pot, and underneath the surface of the thermostatic liquid, was placed an armored resistor C wound in the form of a helical spiral just slipping into the pot. Through this coil was sent an intermittent current which served to maintain the temperature of the thermostat at a constant value. The make and break of the regulating current was effected by means of a constant volume gas thermometer. This thermometer consisted of a thin walled steel tube D, having a diameter of 4 cm. and a length of approximately 15 cm., all joints being welded. This bulb was connected with the electrical contact device by means of a small steel tube E, (Figs. 1 and 2). This tube had a thickness a little under 1 mm. and a diameter of approximately 3 mm.

The regulating device is outlined in Fig. 2. The steel thermometer bulb was attached to tube F of this device by means of a deKhotinsky seal at X. Tube F was connected with a mercury column AB. The height of mercury in this column was adjustable by means of a movable reservoir L and could be fixed by closing the stopcock K. The free space above the mercury column C was exhausted through stopcock G. The regulator, including the thermometer bulb, could be exhausted through stopcocks I and J. Initially, the mercury was lowered in the reservoir L until connection between the arms A and B was established after which the stopcock K was closed. The thermometer bulb was then exhausted through I and filled with nitrogen at a pressure of approximately $1\frac{1}{4}$ atmospheres. Stopcock K was then opened, C was exhausted through G and the mercury level was adjusted to make contact with the platinum point M at the desired temperature in the thermostat. Stopcocks J and I were then closed. The diameter of the tube A was approximately 8 mm. The chamber C, in which was located the upper level of the mercury column, had a diameter of approximately 2 cm. The purpose of this was to increase the sensitivity of the contact device. The free volume of the tube above the mercury surface A was made as small as possible, while the connecting tubes E and F had an internal diameter of about 2 mm. The object of this was to reduce the external volume to a minimum so as to increase the sensitivity as well as to improve the constancy of the regulator. Tube D was a chamber filled with nitrogen in the initial filling. By opening stopcocks H and J, this reservoir could be connected with the thermometer bulb when it was necessary to allow the thermostat to cool down to comparatively low temperatures. To change the temperature setting of the thermostat, the temperature was raised to approximately the desired point, stopcock K was opened and the level of the reservoir L adjusted until contact was made with the platinum point at M. On closing stopcock K, the existing temperature was automatically maintained.

The device above described proved to be extremely convenient and reliable at temperatures from 200°–600°. The temperature of the thermostat was read by means of a platinum, platinum-rhodium thermocouple and regulation was effective to 0.1°, as indicated by the couple. To prevent oxidation of the alloy in the thermostat at higher temperatures, a thin layer of powdered graphite was placed over it.

Conductivity Cells. Two types of conductivity cells were employed depending upon the specific resistance of the mixture. The first type, illustrated in Fig. 3, consisted of a pair of platinum wires having a diameter of about 0.7 mm. and a length of 5 mm. sealed through a Pyrex tube *A*. These wires were joined to a pair of copper leads *F* passing through a rubber stopper *D*. The electrode tube *A* was inserted in a Pyrex tube *B*, into which it fitted closely, closure of the junction being effected by means of a short length of pure gum tubing *C*. The relative position of the tubes was carefully gauged, since the constant of the cell depended slightly upon the position of the electrodes. The interior of tube *A* was connected with the interior of *B* through a small opening *G* in order to prevent the passage of the melted mixture into the interior of *A* along the platinum wires. At the bottom, the electrode tube *A* was provided with a loop of glass rod *E*. The purpose of this was to facilitate the stirring of the mixture which was accomplished by turning the tube *A* through an angle.

These cells were calibrated by comparison with a standard cell of the pipette type. A solution of potassium iodide, nearly saturated with iodine, was employed in making the comparison. The presence of the iodine eliminates polarization and makes it possible to determine the cell constant of unplatinized electrodes. The resistance of the leads was determined by dipping the electrodes into mercury and measuring the resistance by means of a precision bridge. The cell constants and the data from which these constants were derived are given in Table I.

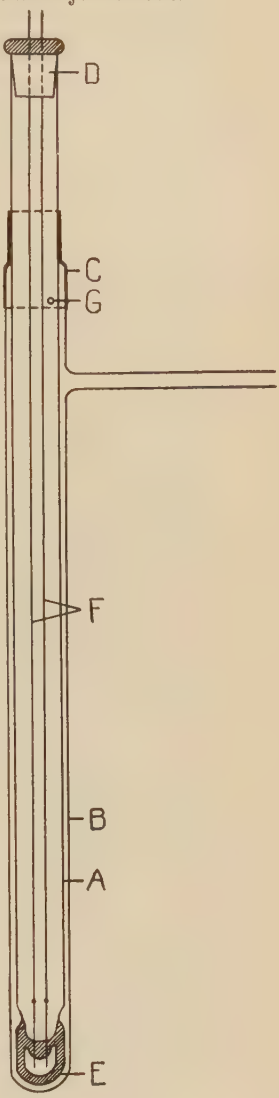

Fig. 3

Cell for measuring conductance of mixtures of S and Te rich in S.

Table I

Lead Resistances and Cell Constants for Cells used in measuring the Resistance of Mixtures rich in Sulphur

Cell No.	R.	Lead R.	R. (Corr.)	Cell Const.
4	296.9	6.9	290.0	3.442
5	141.9	5.4	136.5	1.620
6	216.6	6.1	210.5	2.449
7	207.6	5.1	202.5	2.404
8	175.1	6.0	169.1	2.007
Pipette Cell (comparison)	286.7	0.4	286.3	3.397

For measuring the resistance of tellurium and of mixtures rich in tellurium, it was necessary to use a cell having a high resistance capacity. Such a cell is outlined in Fig. 4. Two arms, *KK* are joined by a length of capillary tubing *AA* having an internal diameter of about 1 mm. Platinum leads *BB*

are joined to copper conductors and sealed into tubes CC. The platinum points are adjusted to make contact with the metal at the ends of the capillary AA. The mixture to be melted is introduced into tube D which connects with the capillary AA by means of tube L. Closely fitting in tube D is a plunger (not shown in the figure) reaching to the bottom of that tube. This plunger can be raised or lowered at will by slipping through a rubber connection at the top of tube D. After introducing the metal into tube D and filling with nitrogen, the cell is placed in the thermostat and, when the material in D is melted, the plunger is lowered to the bottom forcing the melt to pass through tube L and through capillary AA making contact with the electrical circuit through the leads BB. When the measurements have been completed and while the mixture in the capillary AA is liquid, the plunger in D is raised and the melt is blown out of the capillary into tube D by means of a slight pressure of nitrogen on the melt in the tubes KK. This procedure is essential as otherwise it is practically impossible to clean the capillary. The pressure between KK and D is equalized or adjusted by means of stopcock G. The cell must of course be cleaned after each series of measurements. This cell was calibrated by means of a 1.0 N solution of potassium chloride, the specific conductance of which was assumed to be 0.1118 at 25°. The solution was made up by weight according to the procedure of Kraus and Parker.[1]

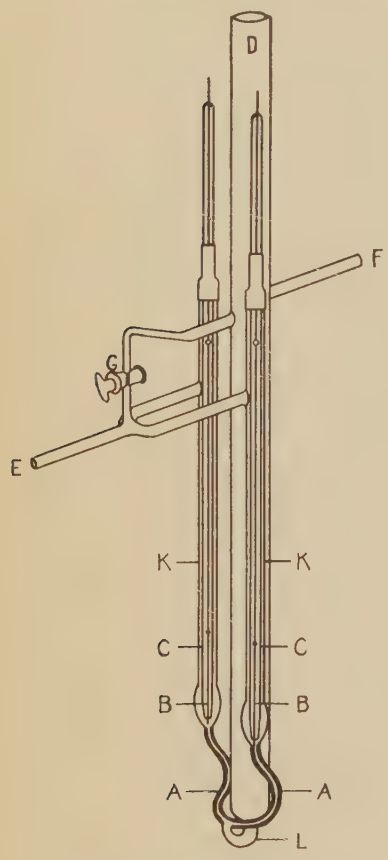

Fig. 4
Cell for measuring conductance of mixtures of S and Te rich in Te.

The cell constants are given in Table II.

Table II

Constants of Cells for Pure Tellurium and Mixtures rich in Tellurium

Cell No.	R.	Cell Const.
1	2303	257.8
2	6340	708.8
3	4435	495.8

For cells 2 and 3, the lead resistance was determined under working conditions by dipping the electrodes into molten tin and determining the resistance at various temperatures. The leads of cell 1 were the same as those of cell 2 except that the length of platinum was shortened. The corrections for cell 1 were calculated from those of cell 2 assuming the value 0.0036 for the resistance temperature coefficient of platinum. The results are given in Table III.

[1] Kraus and Parker: J. Am. Chem. Soc., **44**, 2422 (1922).

TABLE III

Lead Resistance for Cells 1, 2 and 3 at Different Temperatures

Temp. °C.	Resistance		
	Cell 1	Cell 2	Cell 3
349.6	0.3002	0.2935	0.2900
369.0	0.3029	0.2960	0.2923
395.0	0.3065	0.2985	0.2947
416.0	0.3094	0.2999	0.2959
429.2	0.3113	0.3013	0.2960
457.0	0.3152	0.3044	0.2990
486.0	0.3192	0.3070	0.3018

Other Apparatus. The temperature was measured by means of a platinum, platinum-rhodium thermocouple previously calibrated against the melting points of tin, lead, zinc and aluminum and the boiling point of water.

For measuring the resistance of pure tellurium and mixtures of tellurium and sulphur, containing up to 15 at.% of sulphur, a Leeds and Northup precision bridge was used. For mixtures containing 30 or more at.% of sulphur, a Kohlrausch bridge and telephone were employed. With a 30 at.% mixture, the resistance as measured according to the two methods differed by about one-half percent due to polarization effects which were noticeable in the case of the D.C. method. Mixtures containing more than 30 at.% of sulphur showed marked polarization effects.

Procedure. In the case of mixtures rich in sulphur the desired quantities of sulphur and tellurium were weighed out, ground together in an agate mortar and introduced into the cell (Fig. 3). The cell was then evacuated and filled with nitrogen. The thermostat was brought to a temperature somewhat above the melting point of the mixture, whereupon the cell was introduced into the thermostat. When the mixture was melted, the electrodes were lowered into the fused mass and the mixture was stirred until a constant value of the resistance was reached. The resistance of the melt was then measured at a series of temperatures.

Tellurium-rich mixtures were premelted in a Pyrex tube under an atmosphere of nitrogen at a pressure of approximately two atmospheres. Special precautions were observed to avoid the loss of sulphur. When the mass was melted, it was thoroughly shaken and then cooled rapidly. The product was removed, when cold, and introduced into tube *D* of the conductivity cell (Fig. 4). The cell was evacuated and filled with nitrogen and a slow stream of nitrogen was kept passing through the upper part of the cell through tubes *E* and *F* against a slight pressure of mercury in a trap attached to *F*. When the mixture was melted, the plunger was depressed as described above, forcing the melt into the capillary *AA* of the cell, after which the resistance was measured at a series of temperatures.

In the case of pure tellurium, the metal, which had received an additional distillation in vacuo, was introduced into tube *D* of the cell (Fig. 4) and melted under nitrogen. The manipulation was similar to that described above in the

case of the mixtures rich in tellurium. It was necessary to carry resistance measurements out as rapidly as possible since tellurium reacts slowly with the platinum electrodes at higher temperatures.

Tellurium has a marked tendency to undercool so that it was possible to extend the conductance measurements a considerable distance into the metastable region of the liquid phase. On solidifying, a discontinuous change occurs in the resistance. In series 1, the cell was provided with a compara-

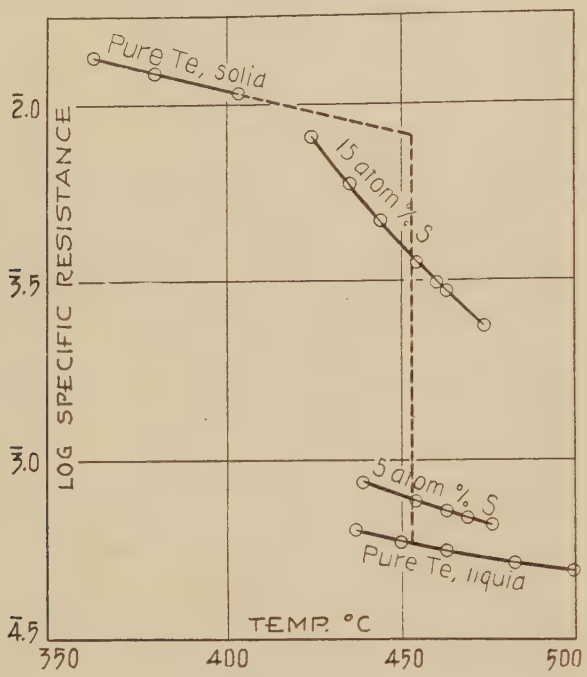

Fig. 5
Resistance of Tellurium and of Sulphur-Tellurium Mixtures

tively large capillary and it was possible to measure the resistance of the solid phase over a considerable temperature range. This was not found possible in the case of cells 2 and 3 which were provided with smaller capillaries, since, owing to contraction which follows solidification, the thread of metal ruptured shortly after solidification occurred.

III. Experimental Results

Cell 1 was used in determining the resistance of pure tellurium in the solid state (Series 1). In this series the values for liquid tellurium are not very precise since the resistance was very low and a small error in the lead resistance caused a relatively large error in the final result. The values for the solid, however, are fairly accurate, since the resistance of this phase is much greater than that of the liquid. More precise data for the conductance of liquid tellurium were obtained in Series 2 in which the resistance of the

capillary was much higher than in Series 1. The results for pure tellurium and for various mixtures of tellurium and sulphur are tabulated below.

TABLE IV

Specific Resistance of Tellurium and of Mixtures of Tellurium and Sulphur

Temp.	R.	R. (Corr.)	Sp. R. × 10^3	
	Series 1. Pure Tellurium (Cell 1)			
480.5	0.4442	0.1257	0.488	
466.0	0.4515	0.1351	0.525	
457.2	0.4575	0.1423	0.553	Liquid
449.1	0.4631	0.1490	0.579	
437.0	0.4762	0.1638	0.636	
431.8	0.4825	0.1708	0.663	
404.7	3.319	3.011	11.69	
380.5	3.732	3.427	13.31	Solid
363.0	4.116	3.814	14.81	
	Series 2. Pure Tellurium (Cell 2)			
499.9	0.6599	0.3513	0.496	
483.1	0.6774	0.3705	0.523	
463.8	0.7045	0.3996	0.564	
450.9	0.7291	0.4255	0.600	
438.0	0.7613	0.4590	0.648	
	Series 3. 5 At.% Sulphur (Cell 3)			
464.0	0.6619	0.3622	0.731	
477.0	0.6341	0.3334	0.673	
469.9	0.6481	0.3479	0.702	Liquid
455.0	0.6862	0.3872	0.781	
440.3	0.7392	0.4415	0.891	
427.5	0.8065	0.5099	1.028	
428.0	7.115	6.818	13.75	
409.5	10.31	10.02	20.21	Solid
382.0	12.06	11.77	23.74	
	Series 4. 15 At.% Sulphur (Cell 3)			
445.3	2.790	2.492	5.026	
461.2	1.948	1.649	3.326	
474.8	1.537	1.236	2.493	
464.0	1.858	1.559	3.145	
455.6	2.182	1.884	3.800	
436.5	3.455	3.158	6.370	
425.5	4.628	4.332	8.738	

TABLE IV (Continued)

Temp.	R.	R. (Corr.)	Sp. R. × 10^3
	Series 5. 30 At .% Sulphur (Cell 3)		
420.0	188.0	187.7	378.6
436.0	131.0	130.7	263.6
457.5	75.4	75.1	151.5
474.1	49.9	49.6	100.0
455.0	81.7	81.4	164.2
444.2	104.5	105.2	212.2
426.0	160.4	160.1	322.9
400.0	294.3	294.0	593.0

Series 6. 50 At.% Sulphur (Cell 3)

Temp.	R.×10^{-3}	Sp. R.×10^{-3}
398.0	16.29	0.0328
421.5	10.90	.0220
440.3	7.44	.0156
469.2	4.01	.0081
455.8	5.35	.0108
440.1	7.58	.0151
423.0	10.81	.0218
416.5	12.31	.0248

Series 7. 70 At.% Sulphur (Cell 4)

Temp.	R.×10^{-3}	Sp. R.×10^{-3}
471.6	3.72	1.08
461.6	4.44	1.29
451.3	5.28	1.53
437.8	6.67	1.94
415.1	10.06	2.92
408.2	11.45	3.33
387.0	16.76	4.87

Series 8. 75 At.% Sulphur (Cell 5)

429.2	31.3	19.3
448.0	21.8	13.5
426.0	32.3	19.9
399.3	54.7	33.8
375.0	88.0	54.3

Series 9. 77.5 At.% Sulphur (Cell 6)

464.1	54.8	21.9
454.9	64.9	26.0
434.1	90.7	36.3
425.5	111.0	44.4
403.9	163.0	65.2
406.1	154.0	61.6
390.1	210.0	84.0
404.1	160.0	64.0

Series 10. 80 At.% Sulphur (Cell 7)

456.1	200.0	83.2
450.8	221.	81.9
429.0	340.	141.0
430.5	323.	134.0
418.0	421.	175.0
405.0	541.	225.0
397.0	655.	272.0
399.3	600.	250.0

Series 11. 85 At.% Sulphur (Cell 8)

394.0	2770.0	1380.0
404.6	1530.	762.
425.5	928.	462.
437.5	482.	240.
444.0	409.	204.
440.1	465.	232.
435.2	530.	264.
428.0	692.	345.
418.1	958.	477.

The results for pure tellurium and mixtures containing 5 and 15 at.% sulphur respectively, are shown graphically in Fig. 5, in which the logarithm of the specific resistance is plotted against the temperature. The corresponding plots for mixtures containing higher percentages of sulphur are similar to those obtained at 5 and 15 at.% except that the curves are steeper and more nearly linear.

From the plots, values of the specific resistance of various mixtures at a temperature of 440° were read off. These values are given in Table V.

TABLE V

Specific Resistance of Mixtures of Sulphur and Tellurium at 440°C

At. % S.	Sp. R.	At. % S.	Sp. R.
0.0	6.410×10^{-4}	70.0	1.870×10^{3}
5.0	8.910×10^{-4}	75.0	1.592×10^{4}
15.0	5.741×10^{-3}	77.5	3.365×10^{4}
30.0	2.390×10^{-1}	80.0	1.145×10^{5}
50.0	1.571×10	85.0	2.317×10^{5}

Discussion

As may be seen from an inspection of Fig. 5, the logarithm of the specific resistance varies approximately as a linear function of the temperature for mixtures of tellurium and sulphur as well as for pure tellurium. With increasing sulphur content the specific resistance increases markedly. This is illustrated in Fig. 6, where the logarithm of the specific resistance is plotted as a function of the composition of the mixture. The resistance of a mixture containing 5 at.% of tellurium does not differ greatly from that of pure tellurium while that of a 15 at.% mixture has a resistance approximately ten times that of pure tellurium at 440°. A mixture containing 85 at.% of sulphur, or $5\frac{2}{3}$ atoms of sulphur per atom of tellurium, has a specific resistance 3.6×10^{8} times that of pure tellurium. Evidently, with increasing sulphur content, the specific resistance of the mixture increases indefinitely and the equivalent conducting power of the tellurium in the mixture approaches a value of zero.

With pure tellurium, as well as with mixtures containing 5 and 15 at.% sulphur, no indications of polarization were observed. Beginning with 30 at.% of sulphur and at higher sulphur concentrations, polarization effects became marked, indicating the presence of ordinary ions. Evidently the electrons due to metallic tellurium associate themselves with sulphur atoms or molecules when the number of atoms of sulphur becomes comparable with that of tellurium. This is not unexpected since the affinity of sulphur for the electrons is fairly high. Since sulphur is a non-polar substance, as is indicated by its low dielectric constant, the ionization of tellurium diminishes rapidly with increasing sulphur content. Evidently the law governing the ionization of tellurium in these mixtures is similar to that governing the ionization of ordinary electrolytes in solvents of very low dielectric constant.

The metallic properties due to tellurium are evidently lost when the number of sulphur atoms present in the mixture becomes equal to or slightly greater than that of the tellurium atoms. As has been pointed out elsewhere,[1] the metallic properties of a substance are greatly dependent on the mean distance between the metallic atoms or ions. If this distance is increased considerably above that of the normal metallic element or compound, the metallic properties disappear.

Fig. 6
Resistance of Sulphur-Tellurium Mixtures at 440°.

It is interesting to note that the specific resistance of both solid and liquid tellurium diminishes greatly with increasing temperature and that a large diminution of resistance occurs as the metal passes from the solid to the liquid state. The ratio of the resistances of the element in the two forms is approximately 1 to 15. It may be noted in this connection that the specific volume of solid tellurium is markedly lower than that of the liquid.

Previous data relating to the specific resistance of pure tellurium are very discordant. Matthiessen[2] gives the value 2.14×10^{-3} for the specific resistance of tellurium at ordinary temperatures while Bridgman[3] gives a value 6.45×10^{-3} for a particular specimen at 24°. Beckmann[4] gives values

[1] Kraus: J. Am. Chem. Soc., **44**, 1218 (1922).
[2] Matthiessen: Pogg. Ann., **103**, 428 (1885).
[3] Bridgman: Proc. Am. Acad. Arts Sci., **52**, 573 (1917).
[4] Beckmann: Physik. Z., **16**, 59 (1915).

ranging from 0.617 to 0.0493 depending on previous treatment. In none of the investigations here cited was the tellurium of known purity. In the present investigation the value found at 263°, the lowest temperature at which measurements were made, is 0.0148 which is much higher than that of Bridgman at ordinary temperatures. Since in the present case, the resistance was found to increase markedly with decreasing temperature, it follows that at ordinary temperatures the specific resistance would probably fall in the neighborhood of the values determined by Beckmann. However, the influence of thermal treatment on specific resistance renders any comparison uncertain if not quite meaningless.

Guntz and Broniewski[1] measured the resistance of a specimen of tellurium through the melting point and found a pronounced maximum for the solid at 50°. Fortsch[2] found the resistance of solid tellurium decreasing regularly from −79 to 280°. Bridgman, as also Beckmann, found the temperature coefficients varying, depending on previous treatment. Excepting the observations of Guntz and Broniewski, already referred to, which are expressed in arbitrary units, no data are available relative to the specific resistance of liquid tellurium.

Summary

Methods and apparatus are described for determining the specific resistance of liquid mixtures of sulphur and tellurium. The specific resistance of pure tellurium has been measured at higher temperatures for both solid and liquid and that of sulphur-tellurium mixtures, up to 85 at.% of sulphur, has been determined over a considerable temperature range for liquid mixtures.

The specific resistance of the mixtures, as also of pure tellurium, decreases with increasing temperature as an exponential function of the temperature. With increasing sulphur content the specific resistance increases greatly. Evidently the equivalent conducting power of tellurium approaches a value of zero with increasing sulphur content.

The specific resistance of liquid tellurium at its melting point is approximately 1/15 that of solid tellurium at the same temperature. Liquid tellurium is, comparatively, a good conductor; at 500° its specific conductance is about 1/6 that of mercury at ordinary temperatures.

The author gratefully acknowledges his indebtedness to the Warren Fund of the American Academy of Arts and Sciences for a grant for the purchase of apparatus which was used in this investigation.

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[1] Guntz and Broniewski: Compt. rend., **147**, 1474 (1908).

[2] Fortsch: Proc. Iowa Acad. Sci., **20**, 523 (1919).